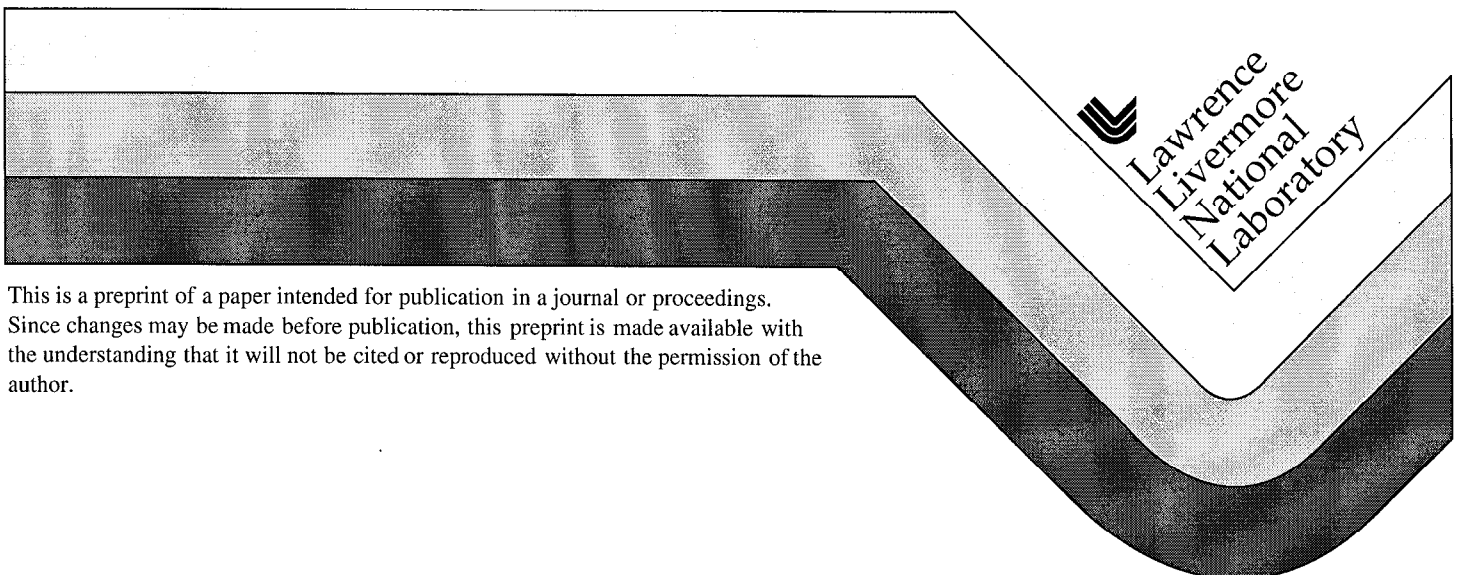


Subsurface Damage Assessment with Atomic Force Microscopy

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Subsurface Damage Assessment with Atomic Force Microscopy

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Introduction

The performance of transparent optics in high fluence applications is often dominated by inhomogeneities in the first few hundred nanometers of material. Defects undetectable with optical methods can cause catastrophic failures when used in critical applications where high strength, chemical or mechanical resistance or extreme smoothness is required. Not only are these defects substantially smaller than the wavelength of visible light, they are often concealed below a layer of glass-like material deposited during the polishing process. In high quality glass, the chemical and material properties of the outermost layer are modified by the grinding, lapping and polishing processes used in fabrication. Each succeeding step in a process is designed to remove damage from the previous operation. However, any force against the surface, no matter how slight will leave evidence of this damage. These processes invariably create dislocations, cracks and plastic deformation in the subsurface region.

In glass polishing, the first 100 nm is comprised of material redeposited from the polishing solution. This redeposition layer is responsible for the extremely smooth surfaces that can be generated on glass. Fortunately for the production of ultra-polished silica based optics, a smooth surface is favored by solution kinetics¹. Unfortunately, the redeposition mechanism brings many unexpected elements into the top layer of the glass. A chemical analysis of the slurry will closely reflect the composition of the first 50 to 150 nm of the surface. Typically, any ions in the polishing slurry are brought into the redeposition layer.

Contamination of the slurry comes from a number of sources including trace components in the abrasive, contaminants in the aqueous base of the polishing solution, residual from previous polishing steps and, for a large part, from the dissolution of the polishing machine.

Inclusion of these contaminant ions generally have a deleterious effect on the physical and chemical properties of the optic reducing performance in high fluence applications and lowering resistance to chemical attack. The modified chemical activity can also be used to strip the surface of the redeposition layer revealing the structure of the region below.

A number of methods can be used to etch the surface, however, the simplest is a timed immersion in a dilute HF bath. The rate of the etching process is a measure of chemical activity and infers stability of chemical bonds. Areas of stress, cracks and dislocations that occur under the redeposition layer, between 100 nm and 500 nm below the surface, will have an etch rate that differs from the bulk. Generally, the greater the stress on a bond, the faster the material will etch. However, in this experiment we have not attempted to use etch rates to assess stability; only to differentiate between different zones in the subsurface

strata of an optic. The effect of force from pre-polishing grinding and lapping steps and from the polishing process itself can be observed once the overlayer is removed. The topography of the surface can be imaged with lateral resolution below 10 nm and a vertical resolution of 0.5 nm by an atomic force microscope (AFM). Subsurface structure can be quantified by a number of techniques, the simplest being the determination of average roughness. The sub-surface structure, inferred from the roughness can be correlated to the distribution of non-native elements below the surface. The dividing line between the redeposition zone and the underlying damage zone can be clearly seen. Further etching beyond the damage layer can determine where the damage ends and the bulk material begins.

Experimental Procedure

High quality fused silica optical flats, using Corning 7980 blanks, obtained from four American and European commercial sources were investigated for subsurface damage. A control group, fabricated at the Lawrence Livermore National Laboratory optical shop, was also tested. The subsurface regions were exposed by a well controlled chemical etch in a dilute HF bath. Using this approach, it is possible to remove as little as a few nanometers of material with a process control of $\pm 5\%$. Material was removed in increments as low as 20nm through the redeposition and damage layer, increasing to 200 nm in the bulk. To prevent excess etching times, a stronger solution was used for the removal of large amounts of material. Samples with the same etch depth from each solution were compared to ensure that each bath was performing as expected.

Secondary ion mass spectroscopy (SIMS) has been used to profile the distribution of elements in the subsurface region. Elements under investigation included aluminum, barium, boron, calcium, cerium, chromium, copper, iron, magnesium, manganese, nickel, sodium and zirconium. By sputtering the surface, depth profiles were obtained for each element up to several microns into the surface. The structure of the subsurface region between 0 nm and 3 microns below the surface in increments determined by the etching step was studied by AFM. Subsurface structure was quantified using several methods of roughness analysis including fractals, power spectral density and grain size along with more traditional methods (Ra and Rrms). Typically, data was collected from 30 and 5 micron square images with 60 and 10 nm resolution per pixel respectively. To avoid distortion of the data from obvious dirt in the sample window, roughness was also determined in a 10 micron area within each 30 micron image. The smaller area was chosen to be representative of the wider area while avoiding features that would skew the data. Each sample was measured a minimum of three times to lend some statistical validity to the measurements.

Results and Discussion

AFM images of three representative depths show a qualitative difference between the surface, a 300 nm etch depth and a 1.5 micrometer etch depth (figures 1-3). Small streaks and scratches, evident on the surface can still be discerned at 300 nm; however, these surface features tend to disappear well before 1 micron. Curiously, with continued etching, new defects will appear, evolve, then shrink away. Typically, these regions are in the shape of nearly round pits. Presence of these features suggests a lower quality optic. In

some of the poorer specimens, the defects link together and are imaged as long tracks (figure 4).

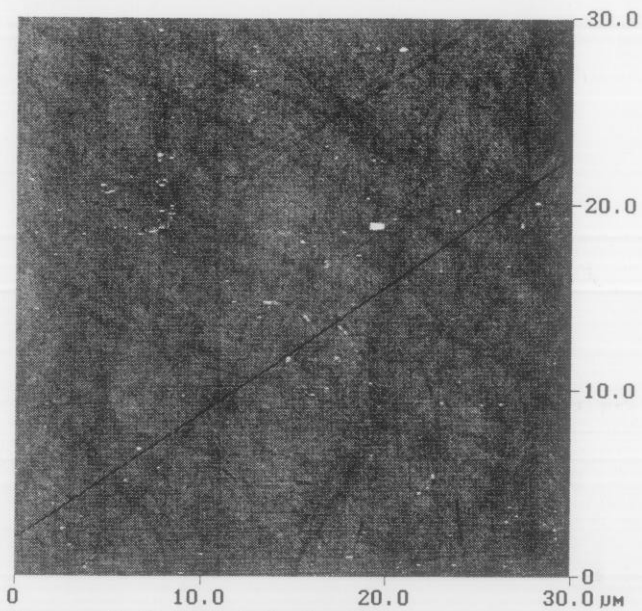


Figure 1: 0 nm etch depth

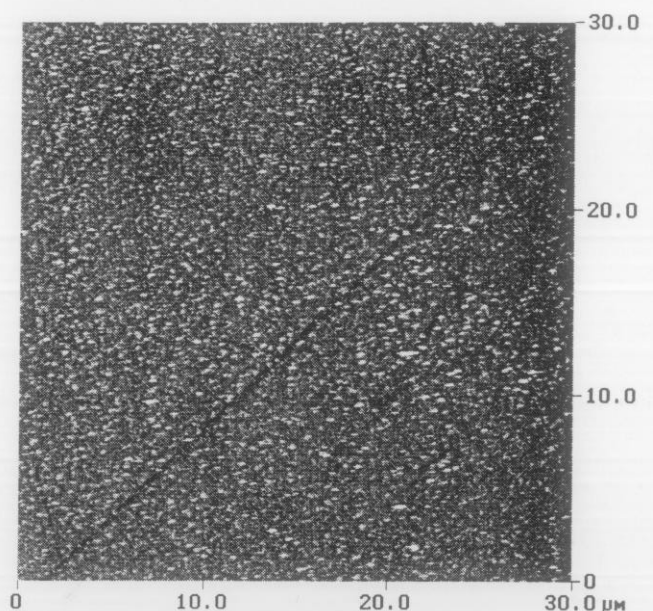


Figure 2: 300 nm etch depth

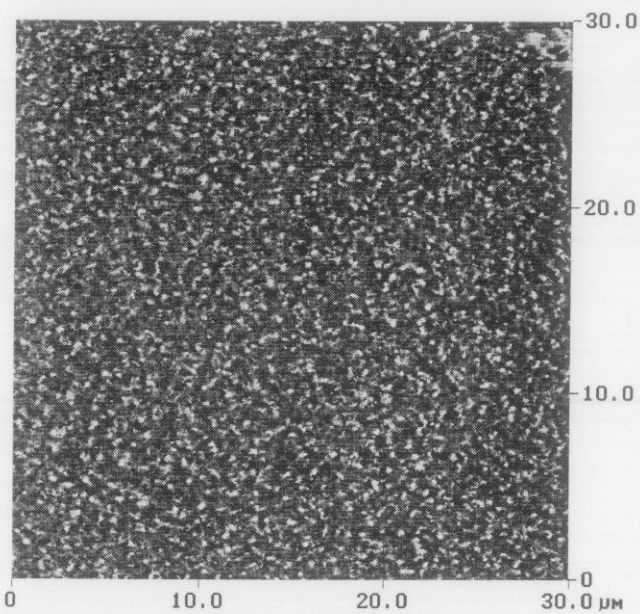


Figure 3: 1.5 micrometer etch depth.

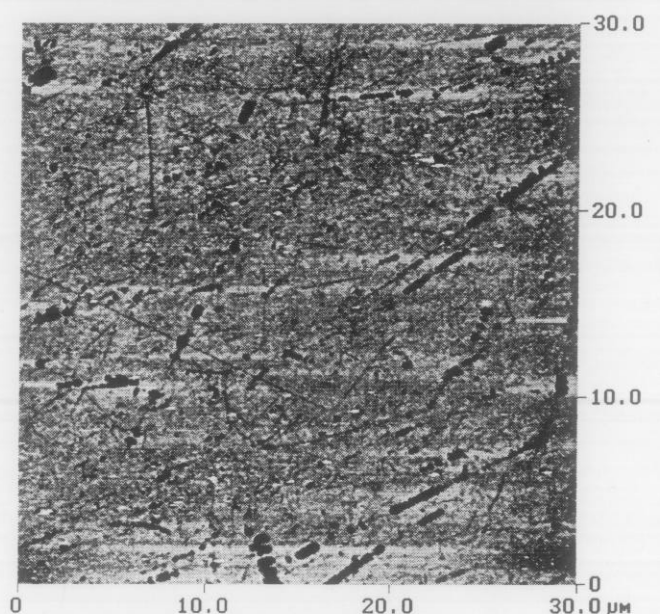


Figure 4: Subsurface defects not apparent at the surface.

A comparison of etch depth for three manufacturers can be found in table 1. While the absolute values are different, each sample follows the same pattern as the etch depth is increased. A sharp increase in roughness is noted as the surface is etched from 0 to 80 nm. Past this region the surface becomes smoother until a subsurface minimum is reached around 120 nm. Further etching causes additional roughness, increasing at a slower rate than the first 80 nm. Prior etching studies reported in the literature have

suggested that etching should cause a surface to decrease in roughness, at least if there are any cracks^{2,3,4}. Clearly, this is not the observation in these experiments.

Etch Depth	Ave. Roughness Manufacturer A in nanometers	Ave. Roughness Manufacturer B in nanometers	Ave. Roughness Manufacturer C in nanometers
0 nm	0.245	0.654	1.025
40 nm	0.422	1.245	1.772
80 nm	0.745	2.051	2.691
120 nm	0.489	1.875	1.803
200 nm	0.688	1.964	2.999
300 nm	0.902	2.516	3.756
500 nm	1.140	3.011	4.781
1000 nm	1.278	3.385	5.264
1500 nm	1.484	3.696	5.840

Table 1: Comparison of roughness for three commercial manufacturers of optical flats

SIMS profiling has indicated that all of the redeposited material occurs within the first 80 nm of the surface. Only the bulk material is present below 80 nm. The redeposition zone, controlled by silica chemistry, was quite constant over the range of samples studied. Correlation between the SIMS and AFM data clearly indicates the division between the redeposition region and a zone of plastic deformation. The depth of the plastic zone was found to be very process dependent, extending from 100nm below to as much as 500 nm in some cases. This is not likely to be the limit as all of our samples were of high quality when compared to standard optical components.

Conclusions

The assessment of subsurface structure by AFM is a promising technique for the determination of process induced distortion. To a certain degree, the history of the process is written in the part. Certain features in the optic can be consistently related to specific manufacturers. While presently focusing on optics, the technique can be used to evaluate process induced damage for many precision components where surface integrity is of interest.

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